Volume 1
Number 12 *Chemical studies of the lime-sulfur lead arsenate spray mixture*

Article 1

1914

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Research Bulletin No. 12 June, 1913

Chemical Studies of the Lime-Sulfur Lead Arsenate Spray Mixture

BY W. E. RUTH

AGRICULTURAL EXPERIMENT STATION
IOWA STATE COLLEGE OF AGRICULTURE AND
THE MECHANIC ARTS

CHEMICAL SECTION

AMES, IOWA

SUMMARY

Mixing lead arsenate with lime sulfur results in:

1. A decrease of sulfur in solution.
2. A decrease of calcium in solution.
3. Increase of thiosulfate in solution.
4. Increase of thiosulfate in the residue.
5. Increase of sulfite in solution.
6. Formation of lead sulfide.
7. Formation of a compound containing arsenic and sulfur but insoluable in the lime-sulfur solution,—probably lead thioarsenate.

CHEMICAL STUDIES OF THE LIME-SULFUR--LEAD ARSENATE SPRAY MIXTURE

BY W. E. RUTH

INTRODUCTORY.

The use of lime-sulfur as an orchard spray originated with M. F. Dusey[1] of Fresno, California. It had been previously used as a sheep dip for killing ticks, but Mr. Dusey tried it successfully as a means of combating San Jose scale. Some years later San Jose scale was discovered in different fruit districts in the middle Atlantic states and after a time the lime-sulfur treatment came to be recognized there, as well as in California, as the best means for combating that insect.

At that time bordeaux mixture was regarded as the most efficient spray for control of fungus diseases in orchards of deciduous fruits, and to control leaf eating insects also, it was the common practice to combine with bordeaux mixture some arsenical poison. It was early recognized that lime-sulfur possessed some fungicidal properties although it had not yet made a place for itself as a standard fungicide. That resulted later through the work of Cordley[2] of Oregon and other investigators.

After numerous experiments in combining lime-sulfur with various arsenical poisons it appeared that lead arsenate was better adapted for this purpose than any other of the common arsenical insecticides. In fact these other poisons when used with lime-sulfur gave deleterious effects as shown in injury to foliage and fruits. The use of lead arsenate as an insecticide was developed during the decade beginning 1892. It was first used by the Gypsy Moth commission of Massachusetts.[3]

The work discussed in this bulletin originated in a project to find, if possible, a substitute for lead arsenate to be used with lime sulfur that would be cheaper, as well as a substitute that would have as valuable combined insecticidal and fungicidal properties as the combined lime-sulfur and lead arsenate mixture. Seeking such a substitute suggested a study of the reaction between lime-sulfur and lead arsenate. This bulletin is a report on the present status of this work.

PREVIOUS WORK.

Much experimental work has been done with arsenicals of various kinds in the way of spraying tests and methods of prep-

[1]Cal. Sta. Bull. 166.
[2]Cordley, Oregon Sta. Bull. 108.
[3]Mass. Hatch Ag. Exp. Sta. Bull. 24.

aration; most of this, however, has been carried on by horticulturists and entomologists. Some work has been done with lime-sulfur, with reference to methods of preparation and the forms of combination in which the sulfur is present in solution, much of it by chemists. As far as known at present Bradley and Tartar[4] have made the only studies of lime-sulfur and lead arsenate mixtures from a chemical standpoint. They compared the neutral with the acid lead arsenate when mixed with lime-sulfur, and the total sulfur and calcium content of the lime sulfur, but made no study of the various forms of sulfur in solution before and after mixing.

EXPERIMENTAL.

COLOR CHANGES

To everyone who has handled lime-sulfur and lead arsenate in a mixture for one spraying, it is well known that a change occurs in the appearance of the lead arsenate. It very soon turns from white to black in presence of lime-sulfur solution. If a solution of lead acetate be added to a solution of sodium thioarsenate, the precipitate when first formed has a reddish yellow color which very soon gives way to a darker brown and this in turn to a very dark gray, almost black.

Several drops of concentrated lime-sulfur solution added to a small quantity of fine particles of lead arsenate suspended in water will result in the lead arsenate undergoing the same color changes described above in the precipitation of the lead thioarsenate. With the addition of the first lime-sulfur solution, the particles of lead arsenate turn to the reddish yellow color and in presence of more lime-sulfur it turns darker and finally black in the excess of lime-sulfur.

Again, if the lime-sulfur be added to the lead arsenate in the water in small diluted portions, up to a certain point the sulfur will be precipitated so as to decolorize completely the solution; beyond that point the color of further additions of lime-sulfur is apparently not affected.

EFFECT OF MIXING ON LEAD ARSENATES.

At first it was attempted to mix small definite quantities of lead arsenate with accurately measured portions of lime-sulfur solution so that definite quantities of lead and arsenate could be accounted for after mixing with lime-sulfur. This, however, was abandoned, because it was found impractical to separate the black lead-arsenate-sulfur residue quantitatively

[4] Vols. I & II Jour. of Ind. & Eng. Chem.

from the filter upon which it was separated and washed from the lime-sulfur.

To avoid this difficulty, quantities of approximately one hundred grams of lead arsenate were mixed with lime-sulfur. (Lime-sulfur concentrate 32.5°B. diluted 1 to 40, about the concentration recommended for summer spray.) The mixture was prepared from Grasselli commercial lime-sulfur and Sherwin-Williams "New Process" lead arsenate which was slightly acid to litmus. The latter showed the following analysis, dry basis:

Total lead (Pb) Per cent	Total arsenic oxide (As_2O_5) per cent	Water soluble As_2O_5 per cent
64.18	26.42	.42
64.28	26.52	.46

The lime-sulfur was used in large excess in an attempt to simulate conditions that obtain in spraying. The mixture was very frequently stirred to prevent the caking that is so common with lead arsenate in lime-sulfur. After standing several hours the lead-arsenate-sulfur residue was filtered with suction and washed with previously boiled and cooled water to the complete disappearance of color of lime-sulfur solution, and then with several hundred c.c. more of water to insure complete removal of lime-sulfur. The residue was dried in an oven not over 40°C. and kept in a vacuum desiccator until ready for analysis.

One gram samples were digested with 100 c.c. of approximately ten per cent nitric acid solution on the steam bath for several hours; filtered, washed and the digestion repeated. The solution contained the lead, arsenic and calcium. It was transferred to a 300 c.c. volumetric flask, diluted to the mark and aliquots used for determining the lead and arsenic. The lead was determined from the solution by the chromate method and arsenic by the modified Gooch and Browning method.

One preparation of the mixture gave the following results for lead and arsenic oxide:

	Lead (Pb) Per cent	Arsenic Oxide (As_2O_5) Per cent
1.	42.61	8.87
2.	42.17	9.06
3.	42.24	8.86
4.	42.28	8.89

A second preparation gave the following by the same method:

Lead (Pb) Per cent	Arsenic oxide Per cent
45.52	9.09
45.72	9.09

The total sulfur, free sulfur and calcium were also determined in the second preparation, showing the following:

Total sulfur Per cent	Free sulfur Per cent	Total Calcium (Ca) Per cent
22.51	19.32	5.14
22.52	19.56	5.02

Total sulfur was determined by the Fresenius[5] method, the arsenic being removed from the hydrochloric acid solution with H_2S. The calcium was determined by the Rose[6] method of decomposition with chlorine and separation of lead and arsenic by hydrogen sulfide. Free sulfur was determined by carbon disulfid extraction.

The decrease in percentages of lead and arsenic from those of the original lead arsenate to the percentages in the mixture is very marked. The decrease is apparently due to the addition of sulfur and calcium to the mixture. But the results of analyses do not total a hundred per cent, as shown below.

Pb—45.62
Total S—22.51
Ca— 5.08
As_2O_5— 9.09

82.30

This evident loss prompted examination of the mixture for the presence of oxygen compounds of sulfur, such as sulfates, sulfites or thiosulfates. Accordingly one gram samples were placed in the shaker bottles with about 800 c.c. of previously boiled and cooled distilled water, then shaken for four hours and filtered. These solutions were made up in liter flasks and aliquots used in analyses. Solutions gave strong tests for calcium with ammonium oxalate, as well as a test for thiosulfate, but no evidence of sulfids, sulfites or sulfates.

The solutions decolorized iodine solution but when bleached again with stannous chloride gave no precipitates with barium chloride in hydrochloric acid solution (even after long standing), thus eliminating presence of sulfites or sulfates. A portion of the solution boiled with hydrochloric acid became cloudy in appearance due to free sulfur precipitating from the decomposition of thiosulfate. Other portions of the solutions mixed with chlorine water and allowed to stand over night, then boiled and acidified with hydrochloric acid gave liberal precipitates of barium sulfate indicating the thiosulfate.

[5]Treadwell & Hall, p. 357, V. II, 3rd ed.
[6]Ibid., p. 359.

Quantitative determinations of sulfur by the latter described method and calculated for the whole solutions gave the following results

	Free Sulfur grams	Calculated equivalent of CaS_2O_3 Grams
1.	.0188	.03324
2.	.0185	.04255
3.	.0176	.04048
4.	.0194	.04462
5.	.0162	.03726
6.	.0159	.03657
7.	.0162	.03726
8.	.0172	.03956

The amounts of calcium thiosulfate shown here calculated to the original weight of sample show from 3.3 to 4.4 per cent thiosulfate present. The solutions showed only traces of arsenic, so that very small quantities of calcium arsenate could possibly be present.

The Cornell Station[7] has shown that mixing lead arsenate with lime-sulfur increases the fungicidal value of the latter. Haywood[8] in his work on lime-sulfur says: "It is a well known fact that sulfites act as antiseptic agents. There is reason to believe that they would also act as insecticides. From the decomposition of the wash there are obtained sulfur in a very finely divided form, thiosulfate for a time, and sulfite which is gradually set free. The writer (Haywood) is of the opinion that these are the active agents in killing insects." Haywood shows by reaction how the thiosulfates are formed from the polysulfides. If it is true, as he supposes, that the fungicidal properties of lime-sulfur are largely due to the formation of thiosulfates and sulfites with liberation of free sulfur, here then is an explanation, partially at least, for the increase of the fungicidal properties of the lime-sulfur solution when mixed with lead arsenate.

Bradley and Tartar[9] say that no sulfids of arsenic are formed since they would be soluble in the alkaline lime-sulfur solution, and none was found in the solution. The writer also failed to find any sulfid of arsenic in the lime-sulfur solution.

If a portion of the lead-arsenate-sulfur residue be covered with a ten per cent sodium hydroxide solution, then warmed on the steam bath for a short time and alowed to settle, some of the supernatant liquid when acidified with hydrochloric acid will be shown to contain arsenic sulfid. A small quantity will be thrown out of solution. A part of the residue remaining

[7]Wallace, Blodgett & Hesler, Cornell Sta. Bull., 290.
[8]Haywood, Bureau of Chem., U. S. D. A., Bull. 101.
[9]loc cit.

after extraction for free sulfur was treated with the alkali, but the solution gave no precipitate of arsenic sulfid when acidified. A second portion of the residue which had been extracted until free from free sulfur was warmed with sodium polysulfid. After standing several hours on the steam bath the liquid gave quite a heavy precipitate of arsenic sulfide when acidified with hydrochloric acid. Arsenic in the arsenic sulfid precipitates was verified by boiling with hydrochloric acid and potassium chlorate and precipitating as magnesium ammonium arsenate.

EFFECT OF MIXING ON LIME-SULFUR.

To observe the effect of mixing on the lime sulfur solution, approximately one gram samples of the lead arsenate, dried free from moisture, were mixed with accurately measured quantities of the lime-sulfur solution (concentrate 32.5°B. used in different dilutions). The mixtures were made in small ground glass stoppered flasks that were so filled by the quantities of lime-sulfur used as to exclude all air possible. The lime-sulfur was separated from the lead arsenate residues by means of Gooch filters. Aliquot parts of ten cubic centimeters were immediately drawn off, and the determinations of total sulfur made from one, and total sulfids, thiosulfate, sulfite and sulfate all made from a second part.

The mono-sulfid sulfur was not determined but total sulfid sulfur was precipitated by iodine solution, determined as barium sulfate and the iodine titration continued for thiosulfate as described by Harris[10].

As comparative results were sought in the lime-sulfur before and after mixing with lead arsenate the above method was found very satisfactory and quick for precipitating the sulfid sulfur.

After the thiosulfate titration the sulfid sulfur was filtered off, and sulfites, now oxidized to sulfates, along with any sulfates originally present, were precipitated as barium sulfate in the cold hydrochloric acid solution by standing at least twelve hours.

The results of analyses of lime sulphur mixed with lead arsenate as described above are shown below in tables I and III Those shown in table I are for mixtures standing one hour and table III shows results for mixtures standing over night. Tables II and IV show the differences in the various forms of sulfur, being more or less as the case may be, than in the original lime-sulfur solution before mixing with lead arsenate.

Tables II and IV show losses of sulfid sulfur and calcium from solutions and increases of thiosulfate and sulfate (including sulfite) in every case. Mixtures Nos. 1, 2, 3, in both tables I and III were made at the same time from the same diluted

[10]Mich. Sta. Tech. Bull. No. 6.

lime-sulfur solution with approximately the same weights of lead arsenate as shown in the tables. The results in the first

TABLE I.

ANALYSES OF LIME-SULFUR SOLUTION WITH LEAD ARSENATE STANDING ONE HOUR. WEIGHS OF VARIOUS FORMS OF SULFUR IN GRAMS.

	1	2	3	4	5	6	7
No.	Sulfid -S-	Sulfate -S-	Thiosulfate -S-	Total S- calculated	Total S- determined	Wt. of lead arsenate used	Wt. of Ca in sol.
1	.31200	.00281	.03477	.34958	.32964	1.0006	.09539
2	.30066	.00309	.03635	.34010	.32031	0.9997	.09333
3	.29922	.00295	.03635	.33852	.32243	1.0013	lost
*Blank 4	.42132	.00116	.01738	.43986	.43735	none	.12116
5	.23245	.00178	.03984	.27407	.24525	1.0128	.07817
6	.20834	.00178	.03824	.24836	.22105	1.0015	.08141
7	.21020	.00178	.03984	.25182	.22222	1.0013	.07434
8	.20697	.00175	.03984	.24766	.22002	0.9996	.07640
9	.21844	.00179	.03984	.25907	.23478	1.0203	.07537
10	.21246	.00178	.03824	.25248	.22846	1.0088	.07611
Blan 11	.38680	.00104	.02231	.41015	.38859	none	.11791

three mixtures table I show that the thiosulfate in solution is increased in the lime-sulfur by mixing as well as the sulfate (including sulfites), but upon further examination of 1, 2, 3, table III, it will be seen that the oxidation has continued—the thiosulfates having decreased and sulfates increased. It must

*The "blank" determination before mixing is for the same quantity of the same lime-sulfur solution used in the mixtures given immediately above.

be borne in mind that the flasks were full to exclusion of air and any oxidation could come only from the lead arsenate, also that the water used in dilution of lime-sulfur concentrate had been previously boiled to free it from air and carbon dioxide.

TABLE II.

DIFFERENCES IN GRAMS IN TABLE I FOR WHOLE AMOUNT OF LIME-SULFUR USED BEFORE AND AFTER MIXING. (1 HR.)

	1	2	3	4	5	6
No.	Sulfid -S- Loss	Sulfate -S- Gain	Thiosulfate -S- Gain	Difference between Total S determined and Total -S- calculated. Gain	Wt. of lead arsenate used	Ca loss from solution
1	.10932	.00165	.01739	.01994	1.0006	.02577
2	.12066	.00193	.01897	.01979	0.9997	.02783
3	.12210	.00179	.01897	.01609	1.0013	lost
5	.15435	.00074	.01753	.02882	1.0128	.03974
6	.17846	.00074	.01593	.02731	1.0015	.03650
7	.17660	.00074	.01753	.0 960	1.0013	.04357
8	.18073	.00071	.01753	.02764	0.9996	.04151
9	.16836	.00075	.01753	.02429	1.0203	.04254
10	.17434	.00074	.01593	.02402	1.0088	.04180

Nos. 5 to 14, table III, are mixtures made with smaller quantities of a more dilute lime-sulfur to observe, if possible, the effect of lesser concentration on the mixture. In table IV it will be noticed that the amount of sulfid sulfur lost from the weaker solution approaches very closely the amount lost from solution in the more concentrated form. The sulfid sulfur loss is due to precipitation in some form by the lead arsenate as well as the formation of thiosulfates and sulfates from the sulfids.

The loss of calcium from solution apparently is due to only partial solubility of the calcium thiosulfate in the lime-sulfur, as well as due to any small quantities of calcium arsenate that

may be formed, the latter being mostly insoluble in the excess of lime-sulfur solution.

TABLE III.

ANALYSES OF LIME SULFUR SOLUTION WITH LEAD ARSENATE STANDING OVER NIGHT. WEIGHTS OF VARIOUS FORMS OF SULFUR IN GRAMS.

	1	2	3	4	5	6	7
No.	Sulfid -S-	Sulfate -S-	Thiosulfate -S-	Total S- calculated	Total S- letermined	Wt. of lead arsenate used	Ca in sol.
1	.21782	.00350	.02687	.24819	.25052	1.0024	.07537
2	.21391	.00419	.02529	.24339	.23925	1.0017	.07493
3	.23390	.00391	.02055	.25836	.26198	1.0021	.08067
*Blank 4	.42132	.00116	.01738	.43986	.43735	none	.12116
5	.0364	.0024	.0231	.0619	.0797	1.0125	
6	.0362	.0020	.0231	.0613	.0770	1.0194	
7	.0352	.0022	.0245	.0619	.0782	1.0122	
8	.0352	.0021	.0191	.0564	.0669	1.0065	
*Blank 9	.2162	.0040	.0127	.2299	.2269	none	
10	.0607	.0031	.0167	.0805	.0860	1.0007	
11	.0688	.0029	.0175	.0892	.0946	0.9994	
12	.0739	.0031	.0159	.0929	.0969	1.0072	
13	.0648	.0032	.0159	.0839	.0917	1.0089	
*Blank 14	.2448	.0040	.0155	.2613	.2680	none	

*The "blank" determination before mixing is for the same quantity of the same lime-sulfur solution used in the mixtures given immediately above.

TABLE IV.

DIFFERENCES IN GRAMS IN TABLE III. FOR WHOLE AMOUNT OF LIME-SULFUR USED BEFORE AND AFTER MIXING. (12 HRS. OR MORE.)

	1	2	3	4	5	6
No.	Sulfid -S- Loss	Sulfate -S- Gain	Thiosulfate -S- Gain	Difference between Total S determined and Total -S- calculated. Loss.	Wt. of lead arsenate used	Ca loss from solution
1	.20350	.00234	.00949	.00233	1.0024	.04579
2	.20741	.00303	.00791	*.00414	1.0017	.04623
3	.18742	.00275	.00317	.00362	1.0021	.04049
5	.1798	.0014	.0104	.0178	1.0125	----------
6	.1800	.0010	.0104	.0157	1.0194	----------
7	.1810	.0012	.0118	.0163	1.0122	----------
8	.1810	.0011	.0064	.0105	1.0065	----------
10	.1841	.0021	.0012	.0055	1.0007	----------
11	.1760	.0019	.0020	.0054	0.9994	----------
12	.1709	.0011	.0004	.0040	1.0072	----------
13	.1800	.0022	.0004	.0078	1.0289	----------

*No. 2 is the only exception in the table where the total sulfur as calculated by summing up the three forms of sulfur showed a gain over the total sulfur as determined.

In columns 4 and 5 tables I and III are given the total sulfurs in the lime-sulfur solutions. The total sulfur as calculated (shown in column 4) is found by summing up the three forms of sulfur in solution.

The total sulfur shown in column 5 is that determined directly on a separate portion of the solutions. Column 4, tables II and IV, show the differences between the two results for total sulfur.

The difference shown in table II is indicated as a gain, that is, the calculated total shows a gain when compared with total sulfur by determination. This difference was at first thought to be due to analytical error but when the same comparisons

were made for the mixtures standing over night, with only one exception as indicated, the calculated totals show a loss as compared with the total sulfur as determined direct.

Whether the gain as shown by the calculated total at the end of an hour is due to analytical error and the loss shown by the calculated total of mixtures after longer standing due to formation of a form of sulfur not included by the present analytical methods, or whether they are both due to analytical error is not understood and no explanation is offered for it at this time.

DISCUSSION OF RESULTS.

As stated before the writer failed to find any arsenic sulfid in the lime-sulfur. But the evidence presented leads the writer to believe that a small amount of the arsenic oxide is converted into the sulfid or that it forms a thioarsenate possibly with lead which would hold it insoluble in the weaker alkaline lime-sulfur solution. The close analogy of the color changes undergone by precipitating lead thioarsenate and that of mixing lead arsenate and lime-sulfur led the writer to look for the formation of some compound containing arsenic sulfid.

The results show that the mixing of lead arsenate and lime-sulfur increases the thiosulfates and sulfites in the solution and the thiosulfates in the residue. If Haywood[11] is correct in his view that the fungicidal value of lime-sulfur is due to the presence of thiosulfate and sulfites, this increase of thiosulfate in lime-sulfur upon mixing with lead arsenate probably explains the increased fungicidal value of the lime-sulfur and lead arsenate mixture.

[11] loc. cit.

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